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IS 8399 (1976): 1, 3-dinitrobenzene [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]

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IS : 8399 - 1976

Indian Standard

SPECIFICATION FOR 1, 3-DINITROBENZENE

UDC 667·28·547·546



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INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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AMENDMENT NO. 1 MAY 2002
TO
IS 8399 : 1976 SPECIFICATION 1, 3-DINITROBENZENE

(*Page 3, clause 0.2, Structural Formula*) — Insert the following below the structural formula:

'(CAS No. 99-65-0)'.

[*Page 4, Table 1, Sl No. (iii), col 2 and 3*] — Substitute the following for the existing:

- a) 1, 2 Dinitrobenzene, percent by mass, *Max* 1.5
- b) 1, 4 Dinitrobenzene, percent by mass, *Max* 0.5

[*Page 4, Table 1, Sl No. (iv)*] — Insert the following after Sl No. (iv):

(1)	(2)	(3)	(4)
v)	Matter insoluble in methanol, percent by mass, <i>Max</i>	0.2	when tested as per IS 8399 : 2001

[*Page 7, clause A-2.3.4(f)*] — Substitute '1, 2-Dinitrobenzene' for '2, 4-Dinitrobenzene'.

(PCD 11)

Indian Standard

SPECIFICATION FOR 1, 3-DINITROBENZENE

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(Continued on page 2)

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IS : 8399 - 1976

(*Continued from page 1*)

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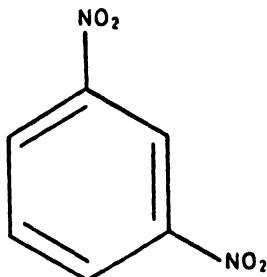
Indian Standard

SPECIFICATION FOR 1, 3-DINITROBENZENE

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 6 August 1976, after the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Chemical Division Council.

0.2 1,3-Dinitrobenzene ($C_6H_4O_4N_2$), also known as *m*-dinitrobenzene, is an important dye intermediate used in the manufacture of dyestuffs. It has the following structural formula:



**I, 3-DINITROBENZENE
(MOLECULAR MASS 168.1)**

0.3 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS:2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for 1,3-dinitrobenzene.

2. REQUIREMENTS

2.1 Description — The material shall be in the form of yellowish brown to brown solid.

*Rules for rounding off numerical values (*revised*).

2.2 The material shall also comply with the requirements given in Table 1.

TABLE 1 REQUIREMENTS FOR 1,3-DINITROBENZENE

SL No.	CHARACTFRISTIC	REQUIREMENT	METHOD OF TEST, REF TO CL NO. IN APPENDIX A
(1)	(2)	(3)	(4)
i)	Crystallizing point, 0°C, <i>Min</i>	88·0	A-1
ii)	Assay, percent by mass, <i>Min</i>	98·0	A-2
iii)	Nitrobenzene, percent by mass, <i>Max</i>	0·2	A-2
iv)	Moisture, percent by mass, <i>Max</i>	1·0	A-3

3. PACKING AND MARKING

3.1 **Packing** — The material shall be packed in steel drums (*see IS:2552-1970**) or as agreed to between the purchaser and the supplier. The containers shall be securely closed.

3.2 **Marking** — Each container shall bear legibly and indelibly the following information:

- a) Name of the material;
- b) Name of the manufacturer and his recognized trade-mark, if any;
- c) Batch number;
- d) Tare, net mass and gross mass; and
- e) The minimum cautionary notice worded as under:

**'STRONG BLOOD POISON. CAUSES CYANOSIS. ABSOLUTELY
AVOID ANY CONTACT WITH SKIN OR DIRECT
INHALATION OF DUST OR FUMES'.**

3.2.1 The containers shall be painted white with red stripes at ends and the cover with skull and bones stencilled in post-office-red colour.

*Specification for steel drums (galvanized and ungalvanized) (*first revision*).

3.2.2 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 Representative samples of the material shall be drawn as prescribed in 3 of IS:5299-1969*.

4.2 Number of Tests

4.2.1 Test for assay and nitrobenzene shall be conducted on each of the individual samples.

4.2.2 Tests for crystallizing point and moisture shall be conducted on the composite sample.

4.3 Criteria for Conformity

4.3.1 For Individual Samples — The lot shall be declared as conforming to the requirements of assay and nitrobenzene if each of the individual test results satisfies the relevant requirement given in Table 1.

4.3.2 For Composite Samples — For declaring the conformity of the lot to the requirements of the characteristics tested on the composite sample (*see 4.2.2*), the test result for each of the characteristics shall satisfy the relevant requirement given in Table 1.

5. TEST METHODS

5.1 Tests shall be carried out according to the methods prescribed in Appendix A, as indicated in col 4 of Table 1.

5.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (*see IS:1070-1977†*) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

*Methods of sampling and tests for dye intermediates.

†Specification for water for general laboratory use (*second revision*).

A P P E N D I X A

(Table 1, and Clause 5.1)

METHODS OF TEST FOR 1,3-DINITROBENZENE

A-1. CRYSTALLIZING POINT

A-1.1 Determine the crystallizing point of the material as prescribed in 7 of IS: 5299-1969*.

A-2. ASSAY AND NITROBENZENE

A-2.0 Outline of the Method — The contents of 1,3-dinitrobenzene and possible impurities, namely, nitrobenzene, 1,2-dinitrobenzene and 1,4-dinitrobenzene are determined by gas chromatographic analysis.

A-2.1 Apparatus

A-2.1.1 Gas Chromatograph — with flame-ionization detector.

A-2.1.2 Potentiometric Strip-Chart Recorder — full scale deflection 1 second.

A-2.1.3 Temperature Programmer

A-2.1.4 Column — made of glass, 1·5 m long, 6 mm in diameter, and filled with 20 percent XE-60 on Chromosorb W (AW-HMDS)† or an equivalent material.

A-2.2 Reagents

A-2.2.1 Acetone — solvent.

A-2.2.2 1,3-Dinitrobenzene

A-2.2.3 1,2-Dinitrobenzene

A-2.2.4 1,4-Dinitrobenzene

A-2.2.5 4-Nitrotoluene — internal standard.

A-2.2.6 Nitrobenzene

A-2.3 Procedure

A-2.3.1 Operating Parameters of Gas Chromatograph

A-2.3.1.1 Injection port temperature — 250°C.

A-2.3.1.2 Column temperature — 160°C.

*Methods of sampling and tests for dye intermediates.

†These are imported chemicals and are generally available with the fabricators of gas chromatographs and also with the stockists of chemicals.

A-2.3.1.3 Carrier gas — nitrogen, 30 ml/min.

A-2.3.1.4 Chart speed — 10 mm/min.

A-2.3.2 Sample — Dissolve 1 g of the sample in acetone. Add a known mass of internal standard and make up the volume to 50 ml with acetone. Inject 1 microlitre.

A-2.3.3 Calibration Factor — Internal standardization is the method used for calibration of the results of analysis. Make up and chromatograph known mixtures of component 'n' for which the calibration factor is to be determined together with the internal standard plus other components of the sample. Calculate the calibration factor as follows:

$$K_n = \frac{X_n \times A_s}{X_s \times A_n}$$

where

K_n = calibration factor for component 'n' against internal standard 's',

X_n = mass percent of component 'n' in the sample,

A_s = peak area for internal standard 's',

X_s = mass percent of internal standard 's' in the sample, and

A_n = peak area for component 'n'.

A-2.3.4 Interpretation of Chromatogram — Elution order of components is as follows:

- a) Acetone (solvent used),
- b) Nitrobenzene,
- c) 4-nitrotoluene (internal standard),
- d) 1,4-Dinitrobenzene,
- e) 1,3-Dinitrobenzene, and
- f) 2,4-Dinitrobenzene.

Identification is done with the aid of test mixture.

A-2.4 Calculation

$$\text{Component 'n' in the sample, } = \frac{K_n \times A_n \times X_s}{A_s} \text{ percent by mass}$$

where

K_n = calibration factor for component 'n' (see A-2.3.3),

A_n = peak area for component 'n',

X_s = mass percent of internal standard added to the sample,
and

A_s = peak area for internal standard 's'.

A-3. MOISTURE

A-3.1 Determine the moisture content of the material as prescribed in **9.2** of IS : 5299-1969*.

*Methods of sampling and tests for dye intermediates.

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